National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2261

Chlorinated Pesticides in Hexane
(Nominal Concentration 2 μg/mL)

This Standard Reference Material (SRM) is intended primarily for use in the calibration of gas chromatographic instrumentation used for the determination of the certified compounds. This SRM is a solution of 15 chlorinated pesticides in hexane. Certified concentrations are provided for 14 of the 15 compounds. A unit of SRM 2261 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentrations of Constituent Pesticides: The certified concentrations and estimated uncertainties for 14 of the 15 pesticides are given in Table 1. These values are based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. Table 2 summarizes the calculated and chromatographically determined concentrations. The Chemical Abstracts Service (CAS) Registry Number and Nomenclature of each pesticide component and a representative chromatogram from the GC analysis of the original solution are shown in the Appendix.

NOTICE AND WARNING TO USERS

Handling: This material contains chlorinated pesticide compounds, many of which have been reported to have toxic, mutagenic and/or carcinogenic properties, and should be handled with care. Use proper for disposal methods.

Expiration of Certification: The certification of SRM 2261 is valid, within the measurement uncertainty(ies) specified, until **01 October 2008**, provided the SRM is handled in accordance with instructions given in this certificate. This certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10 °C and 30 °C.

Instructions for Use: Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified concentrations in Table 1 to be valid within the state uncertainty. Because of the volatility of hexane, certified values are not applicable to material stored in ampoules that have been opened for more than three minutes, even if they are resealed.

Preparation and original analytical determinations were performed by R.M. Parris and F.R. Guenther of the NIST Analytical Chemistry Division.

The coordination of the technical measurements leading to the original certification was under the direction of R.M. Parris and W.E. May of the NIST Analytical Chemistry Division.

Conformation analysis and coordination of stability measurements leading to updated certification in 1998 was under the direction of M.M. Schantz.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 06 August 2003

John Rumble, Jr., Chief
Measurement Services Division

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Statistical analysis was provided by S.B. Schiller of the Statistical Engineering Division.

Partial support for the preparation and certification of this Standard Reference Material was provided by the National Oceanographic and Atmospheric Administration, National Ocean Service, Office of Oceanography and Marine Assessment.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by T.E. Gills and B.S. MacDonald of the NIST Measurement Services Division.

Table 1. Certified Concentrations of Chlorinated Pesticides in SRM 2261

Compound[a]	Concentration[b] (mg/kg)	Concentration[b] (μg/mL)[c]
Hexachlorobenzene	3.005 ± 0.014	1.968 ± 0.009
gamma-HCH	3.012 ± 0.015	1.972 ± 0.010
Heptachlor	3.020 ± 0.023	1.977 ± 0.015
Heptachlor epoxide	3.020 ± 0.026	1.977 ± 0.017
cis-Chlordane	3.012 ± 0.019	1.972 ± 0.012
trans-Nonachlor	3.034 ± 0.022	1.986 ± 0.014
Dieldrin	3.012 ± 0.020	1.972 ± 0.013
Mirex	3.041 ± 0.042	1.991 ± 0.028
2,4′-DDE	3.019 ± 0.026	1.976 ± 0.017
4,4′-DDE	3.019 ± 0.015	1.976 ± 0.010
2,4′-DDD	3.013 ± 0.026	1.973 ± 0.017
4,4′-DDD	3.043 ± 0.042	1.992 ± 0.027
2,4′-DDT	2.993 ± 0.014	1.959 ± 0.009
4,4′-DDT	3.004 ± 0.018	1.967 ± 0.012

[a] The CAS Registry Number and CAS Nomenclature of each pesticide component is listed in the Appendix of this certificate.

[b] The certified value is the equally weighted mean of the gravimetric and average chromatographic concentrations. The uncertainty of the certified value is the half-width of a 95 % confidence interval for the mean, with an allowance for systematic error between the methods.

[c] The values listed in μg/mL units were obtained by multiplying the certified value in μg/g (prior to rounding) by the density of the SRM solution at 22.5 °C (0.6547 g/mL). These concentrations are for use in the temperature range of 20 °C to 25 °C and an allowance for the change in the density over this temperature range is included in the uncertainties.

PREPARATION AND ANALYSIS

Pesticides used in the preparation of this SRM were donated by the U.S. EPA Pesticides & Industrial Chemicals Repository, Research Triangle Park, NC and the Office of Reference Materials, Laboratory of the Government Chemist (formerly National Physical Laboratory), United Kingdom. The pesticide solution was prepared at NIST by weighing and mixing the individual pesticides with hexane and mixing until completely dissolved and homogenized. The total mass of this solution was then measured. The calculated concentration based on the mass of the pesticide compound (adjusted for its consensus purity estimate) in the total mass of the solution is given in Table 2 for 14 of the components. The consensus purity estimations of the chlorinated pesticide components were based on NIST analyses using capillary gas chromatography with flame ionization detection, the purity assay information from the component suppliers, and, where appropriate, differential scanning calorimetry. This bulk solution was then chilled to approximately -5 °C and 1.2 mL aliquots were dispensed into 2 mL amber glass ampoules that were then flame sealed.

Aliquots from eight randomly selected ampoules were analyzed in duplicate by gas chromatography with electron capture detection of an immobilized nonpolar stationary phase capillary column, The four polychlorinated biphenyl (PCB) congener internal standards added to each sample for quantification purposes were PCBs, 82, 66, 105, and 180 [1] Calibration solutions consisting of weighed amounts of NIST SRM 1492 (Chlorinated Pesticides in Hexane) and the hexane solution of internal standard compounds were chromatographically analyzed to determine analyte response factors. The chromatograhically determined concentrations of 14 of the compounds are also given in Table 2.

During stability testing in August 1998, the aldrin content was found to be lower than originally certified. Therefore, the certified concentration of aldrin has been removed from the certificate and because of its observed instability a new value is not provided. A capillary gas chromatogram is shown in Figure 1 on page 4.

Table 2. Summary of Calculated and Chromatographic Results[a]

Compound	Concentration	
	Calculated[b] (mg/kg)	GC/ECD[c] (mg/kg)
Hexachlorobenzene	3.00	3.01 (0.03)
gamma-HCH	3.00	3.02 (0.02)
Heptachlor	3.01	3.03 (0.03)
Heptachlor epoxide	3.01	3.03 (0.02)
cis-Chlordane	3.00	3.02 (0.03)
trans-Nonachlor	3.03	3.04 (0.03)
Dieldrin	3.00	3.02 (0.03)
Mirex	3.01	3.07 (0.03)
2,4′-DDE	3.00	3.04 (0.03)
4,4′-DDE	3.01	3.02 (0.03)
2,4′-DDD	3.00	3.03 (0.03)
4,4′-DDD	3.01	3.08 (0.03)
2,4′-DDT	2.99	2.99 (0.03)
4,4′-DDT	3.01	3.00 (0.03)

[a] The summary of results given above is presented for use only as background information. These values are not certified.
[b] Calculated concentration based on the mass of the pesticide (adjusted for consensus purity estimate, see text) in the total mass of the solution.
[c] Concentrations determined by using gas chromatography with electron capture detection. The uncertainties, listed in parentheses, are one standard deviation of a single measurement and only represent within-method variability.

REFERENCE

[1] Ballschmiter, K.; Zell, M.; *Fresenius Z. Anal.*; Chem. Vol. 302, pp. 20-31 (1980).

Certificate Revision History: 06 August 2003 (This revision reflects an updated expiration date); 10 December 1998 (Removal of aldrin certification); 10 January 1992 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

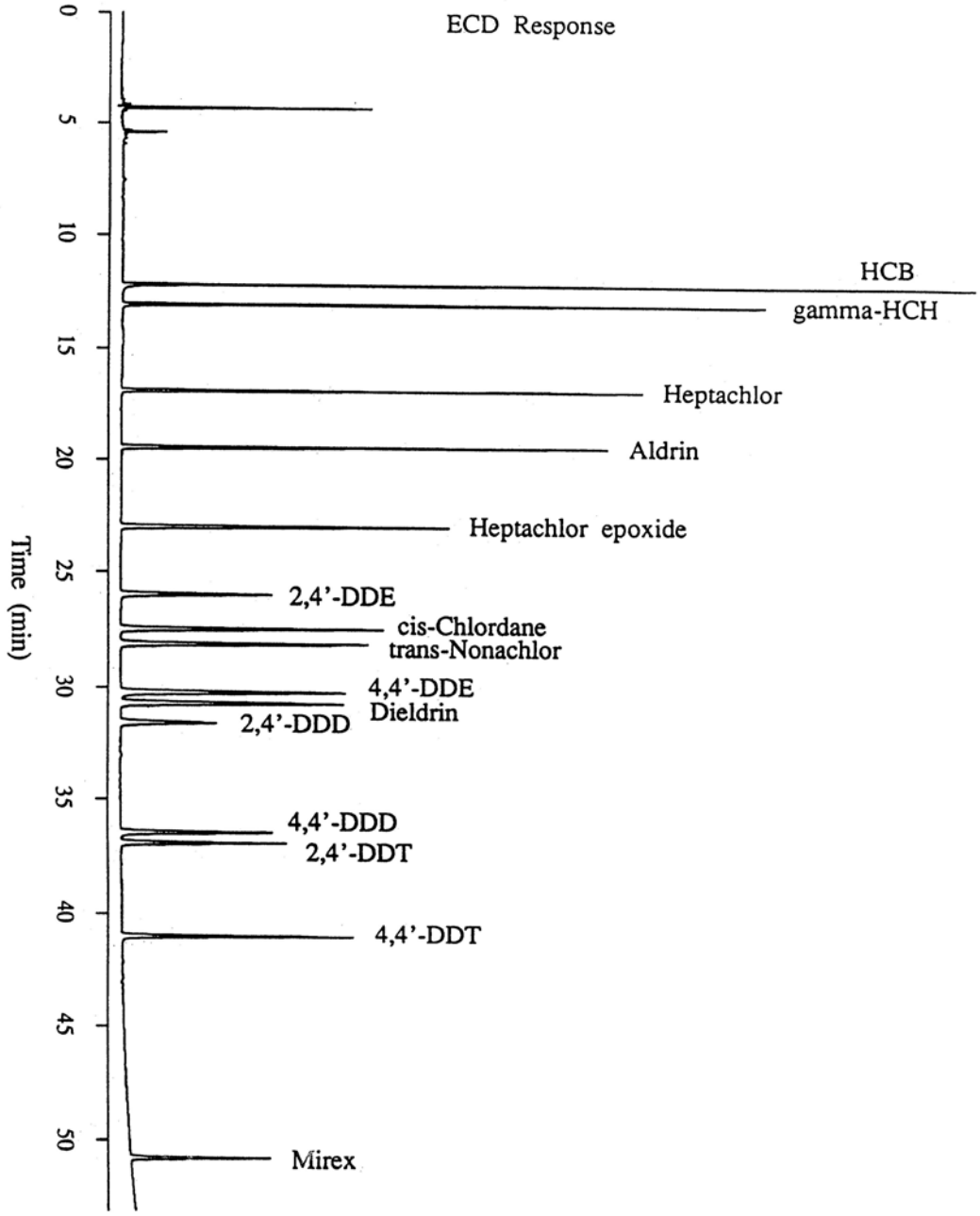

Figure 1. Capillary Gas Chromatogram of NIST SRM 2261.

The following supplementary information may be of interest in connection with the use of this SRM and is supplied for the convenience of the user.

Table A-1. Names, Chemical Abstract Service (CAS) Registry Numbers and Nomenclature[a]

Compound (Alternative Name)	CAS Registry No.	CAS Nomenclature
Hexachlorobenzene (HCB)	118-74-1	hexachlorobenzene
gamma-HCH (gamma-BHC) (Lindane)	58-89-9	(1α,2α,3β,4α,5α,6β)-1,2,3,4,5,6-hexachlorocyclohexane
Heptachlor	76-44-8	1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methano-1H-indene
Aldrin (HHDN)	309-00-2	(1α,4α,4aβ,5α,8α,8aβ)-1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-dimethanonaphthalene
Heptachlor epoxide	1024-57-3	(1aα,1bβ,2α,5α,5aβ,6aα)-2,3,4,5,6,7,7-heptachlor-1a,1b,5,5a,6,6a-hexahydro-2,5-methano-2H-indeno[1,2-b]oxirene
cis-Chlordane (alpha-Chlordane)	5103-71-9	(1α,2α,3aα,4β,7β,7aα)-1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro-4,7-methano-1H-indene
trans-Nonachlor	39765-80-5	(1α,2β,3α,3aα,4β,7β,7aα)-1,2,3,4,5,6,7,8,8-nonachloro-2,3,3a,4,7,7a-hexahydro-4,7-methano-1H-indene
Dieldrin (HEOD)	60-57-1	(1aα,2β,2aα,3β,6β,6aα,7β,7aα)-3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-2,7:3,6-dimethanonaphth[2,3-b]oxirene
Mirex (Dechlorane) (Perchlordecone)	2385-85-5	1,1a,2,2,3,3a,4,5,5a,5b,6-dodecachlorooctahydro-1,3,4-metheno-1H cyclobuta[cd]pentalene
2,4'-DDE (*o,p*'-DDE)	3424-82-6	1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethenyl]benzene
4,4'-DDE (*p,p*'-DDE)	72-55-9	1,1'-(dichloroethenylidene)bis[4-chlorobenzene]
2,4'-DDD (*o,p*'-DDD) (*o,p*'-TDE)	53-19-0	1-chloro-2-[2,2-dichloro-1-(4-chlorophenyl)ethyl]benzene

4,4'-DDD (*p,p*'-DDD) (*p,p*'-TDE)	72-54-8	1,1'-(2,2-dichloroethylidene)bis[4-chlorobenzene
2,4'-DDT (*o,p*'-DDT)	789-02-6	1-chloro-2-[2,2,2-trichloro-1-(4-chlorophenyl)ethyl]benzene
4,4'-DDT (*p,p*'-DDT)	50-29-3	1,1'-(2,2,2-trichloroethylidene)bis[4-chlorobenzene]

[a] Chemical Abstract, Eleventh Collective Index, Index Guide, American Chemical Society, Columbus, OH, (1986).